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### Experiment 1: Melting point of a solid by using Melting Point Instrument

**Aim:** To determine the melting point of given solid substance using melting point instrument

**Requirements:** Given Solid Substance, Capillary Tube, Melting point instrument, Laboratory thermometer.

**Theory:** The change from solid to liquid state of a compound in heating is called melting and the temperature at which a solid in its pure form melts is called the melting point. Every pure solid has a characteristics melting point therefore determination of melting point helps in identification of the compound. Presence of impurities lowers the melting point of the solid. Thus Melting point also serves as a criterion of purity of a compound.

**Procedure:**

1. Take a fine capillary of length 5-6cm. seal its one end by inserting the end of the capillary tube horizontally into the extreme edge of a small steady Bunsen flame for a few seconds, rotating the capillary mean while.
2. Take a small quantity of the compound whose melting point is to be determined on a porous plate and powder it with a spatula.
3. Introduce the powdered compound in the capillary tube by introducing the open end of the capillary tube into the powdered compound and gently rotating it.
4. Gently tap the capillary tube against the porous plate so that the compound sinks into the closed end. Repeat the procedure of introducing and tapping three to four times.
5. Place the thermometer with the capillary tube in the melting point instrument.
6. Start the instrument and finally note down the temperature at which the compound starts melting and completely melts.

**Result:**

Melting point of the given organic compound:

1 .....<sup>0</sup>C

Experiment 2: Determination of pH of beverages using pH meter.

**Aim:** To determine pH of beverages using pH meter.

**Given:**

1. Buffer solutions of pH=4, pH=9.2
2. Beverages

**Procedure:**

1. Standardize the pH meter using buffer solution of pH=4 and 9.2
2. Dip the combined glass electrode of the pH meter in the sample and determine the pH.

**Observation Table:**

Sr. No.	Sample solution	pH of the solution
1.	Coca Cola	
2.	Sprite	
3.	Nimbooz	
4.	Kokam Juice	
5.	Milk	

**Results & Conclusion:**

Experiment 3: Determination of concentration of a given sample of  $\text{KMnO}_4$  by colorimetric method

**Aim:** To determine the concentration of a given sample of  $\text{KMnO}_4$  by colorimetric method

**Requirements:** 1000ppm  $\text{KMnO}_4$  solution, 100  $\text{cm}^3$  standard measuring flasks, 10 $\text{cm}^3$  graduated pipette

**Procedure:**

1. Switch on the colorimeter instrument.
2. Prepare 100ppm solution from the supplied 1000ppm solution (10 $\text{cm}^3$  diluted to 100 $\text{cm}^3$ ). This is the *working standard*.
3. From the working standard prepare a series of solutions as per the following table:

Sr. No.	Volume of 100 ppm solution ( $\text{cm}^3$ )	Concentration of $\text{KMnO}_4$ solution (ppm)	Final volume ( $\text{cm}^3$ )
1	02	02	100
2	04	04	100
3	06	06	100
4	08	08	100
5	10	10	100

4. Take 10 ppm solution and find out the maximum absorbance by taking reading using different filters of the colorimeter.
5. Note down the filter at which the solution gives maximum absorbance. This is  $\lambda_{\text{max}}$ .
6. Record the absorbance of all the other solutions at  $\lambda_{\text{max}}$  in increasing concentration.
7. Dilute the given sample solution upto 100  $\text{cm}^3$  with distilled water in standard measuring flask. Record the absorbance of this solution.
8. Plot a graph of absorbance vs concentration.
9. From the graph, find concentration of unknown sample.

**Observation Table:**

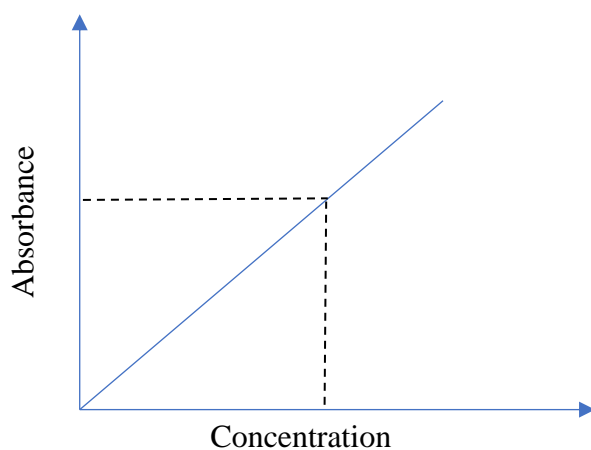
Selection of  $\lambda_{\text{max}}$

Sr. No.	Wavelength	Absorbance


Sr. No.	Concentration of $\text{KMnO}_4$ solution ( $\text{cm}^3$ )	Absorbance
1	02	
2	04	
3	06	
4	08	
5	10	
6	unknown	

**Graph:**

Plot a graph of absorbance against concentration of  $\text{KMnO}_4$

**Calculations:**

From the graph, concentration of  $\text{KMnO}_4$  in unknown solution is  $C_x =$  \_\_\_\_\_ ppm

**Result:**

Concentration of  $\text{KMnO}_4$  in unknown solution = \_\_\_\_\_ ppm

## MICROSCALE EXPERIMENTS IN CHEMISTRY

### INTRODUCTION:

The rising cost of chemicals and decreased flow of funds are causing great concerns to the chemistry teachers. A tug - of - war had also been going on in recent years, to balance the budget of running practical courses and the standards of experiments to be carried out by the students. Since the economy is always the winner, the number of experiments had been the losers. A group of scientists from University of Pune and Fergusson College, Pune are striving hard to maintain the standards of experiments at a friendly budget i.e., by carrying out the organic reactions in capillaries / tiles, using semimicro test tubes for heating experiments (instead of the conventional test tubes) and adopting to microscale preparations. Their attempts serve many purposes.

- (i) cuts down the cost of chemicals.
- (ii) experiments have become environment friendly.
- (iii) less time consuming
- (iv) less hazardous to the teachers, students and lab, assistants. What more it becomes affordable.

### ANALYSIS OF AN ORGANIC COMPOUND

#### A) Preliminary Tests:

EXPERIMENT	OBSERVATION	INFERENCE
<b>1. Test For Aromaticity:</b> a. Substance is introduced into the flame using a nickel spatula.	i. Burns with a smoky flame ii. Burns with a non smoky flame	i. Presence of an aromatic compound ii. Presence of an aliphatic compound
b. Substance is added to a mixture of 3 drops of con. sulphuric acid & 3 drops of con. nitric acid and warmed on a water bath for about 10 minutes. The solution is poured, then into water.	An yellow solution or precipitate is formed	Presence of an aromatic compound The yellow colour is due to the formation of nitro compounds by the nitration of aromatic compounds.
<b>2. Test For Unsaturation</b> a. A drop of the substance is taken on a porcelin tile and add adrop of con. Bromine water to it.	i. Decolouration ii. Decoloration followed by tubidity	i. Presence of unsaturation ii. Presence of aniline/ phenol
b. To a pinch of substance add, a drop of dil. $\text{KMnO}_4$	Decolouration	Presence of unsaturation/ easily oxidisable compound
<b>3. Solubility Tests:</b> i. water	i. Soluble	i. Presence of urea, carbohydrates etc.

ii. 5 % Na <sub>2</sub> CO <sub>3</sub> iii. 5% NaOH iv. 5% HCl	ii. Soluble iii. Soluble iv. Soluble	ii. Presence of acids iii. Presence of acids, phenols iv. Presence of amines
<b>5. Action on Litmus:</b> Moistened litmus paper is brought into contact with the substance	i. Blue litmus paper turns red ii. Red litmus paper turns blue iii. Neutral	i. Presence of acids, phenols ii. Presence of amines iii. Presence of carbohydrates, esters, carbonyls etc.
<b>6. Action of Sulphuric Acid</b> 5 mg. of the substance is warmed with 3 drops of con. H <sub>2</sub> SO <sub>4</sub>	Chars with a smell of burnt sugars	Presence of carbohydrate

**B) Solubility test**

EXPERIMENT	OBSERVATION	INFERENCE
1. About 5mg of the compound is taken on a watch glass. One or two drops of water are added.	a. The compound partially dissolves.  b. The mixture remains undissolved	Presence of carbohydrates, urea amine salts, salts of carboxylic acids, sulphanilic acid dicarboxylic acid etc  b. Absence of all the above
2. In another watch glass 5mg of compound is taken and then tested with a drop of the saturated bicarbonate solution	Brisk effervescence. The solid acid reappears on addition of a drop of 8N HCl	Presence of an acid confirmed.

Theory:

*The carboxylic acids react with bicarbonate to form sodium salts of the acids. Alkali metal salts being water soluble comes along with the aqueous layer. On acidification the free acid is generated as a solid.*



3. In a watch glass 5mg of compound is taken and then	Solubility observed and a Turbidity or oil is formed	presence of phenolic compound confirmed.
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tested with a drop of 2N sodium hydroxide solution.	upon adding a drop of 8N HCl	
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Theory:

*Phenolic compounds react with sodium hydroxide to form sodium phenolate. Since alkali metal salts are soluble in water, phenolate ion comes along with the aqueous layer. Upon acidification, liberates the phenol.*



4. In another watch glass 5 mg of compound is taken and then tested with a drop of 2N hydrochloric acid solution.	Solubility observed and a Turbidity or oil is formed upon adding a drop of 10N NaOH.	presence of amino compound confirmed.
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Theory:

*The amines form amine hydrochlorides with hydrochloric acid. The hydrochloride being water soluble comes along with the aqueous layer. Upon neutralization the free amine is liberated.*



If all the above tests are negative then the given compound is **Neutral** in nature.

### C) Detection of C, H, O, N, S and X<sup>-</sup>

(Lassaigne's Test)

Preparation of Sodium fusion Extract:

Two small cut pieces of sodium are fused in a semi-micro hard glass tube. About 5mg of the substance is added to it and fused again. The tube is cooled to room temperature. Keeping the tube in a slanting position 4 drops of water are added (the first drop of water is allowed to react with excess of sodium. The second drop is added after the initial reaction is over. Then the third and the fourth drops of water are added slowly). This is the sodium fusion extract. The following tests are performed with it.

<b>a. Test for Nitrogen</b> A drop of the extract is placed on a tile. A drop of a con. solution of FeSO <sub>4</sub> followed by a drop of 50% H <sub>2</sub> SO <sub>4</sub> is added to it.	Prussian blue colour is obtained	Presence of nitrogen
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<p><b>b. Test for Halogens:</b> A drop of the extract is treated with con. <math>\text{HNO}_3</math> followed by a drop of <math>\text{AgNO}_3</math> solution</p>	<p>i. Curdy white precipitate soluble in ammonium hydroxide ii. Pale yellow precipitate sparingly soluble in ammonium hydroxide iii. Yellow precipitate insoluble in ammonium hydroxide</p>	<p>i. Presence of chlorine ii. Presence of bromine iii. Presence of iodine</p>
<p><b>If halogen is present,</b> About 5 mg of the substance is warmed with 3 drops of alcoholic <math>\text{AgNO}_3</math> on a water bath and acidify with a drop of con. <math>\text{HNO}_3</math>.</p>	<p>i. Immediate formation of a precipitate ii. No precipitate</p>	<p>i. Presence of halogen in the side chain ii. Presence of halogen in the aromatic nucleus</p>
<p><b>c. Test for Sulphur:</b> A drop of the extract is mixed with a drop of sodium nitroprusside on a tile.</p>	<p>Violet colour</p>	<p>Presence of sulphur</p>

#### D) Determination of class of compound:

##### I. Under C, H, (O) group:

<p><b>1. Test for carboxylic acid:</b> 1a. About 5 mg of the substance is treated with a drop of highly saturated <math>\text{NaHCO}_3</math> solution on a tile</p>	<p>Brisk effervescence</p>	<p>Presence of carboxylic acid</p>
<p>1b. A drop of the substance is mixed with a drop of neutral <math>\text{FeCl}_3</math> solution on a tile</p>	<p>i. Violet color ii. Flesh color</p>	<p>i. Presence of salicylic acid ii. Presence of phthalic acid</p>
<p><b>2. Test for ortho - dicarboxylic acid:</b> About 5 mg. of the substance is heated with an equal amount of resorcinol and 1 drop of con. <math>\text{H}_2\text{SO}_4</math> in a semi micro tube. Take a drop of it at the tip of a glass rod and dip it in very dil. <math>\text{NaOH}</math> in a semi micro tube.</p>	<p>Green fluorescence</p>	<p>Presence of o- dicarboxylic acid</p>

<p><b>3. Test for Phenol:</b></p> <p><b>a. Neutral Ferric chloride test</b> A drop of the substance is mixed with a drop of neutral <math>\text{FeCl}_3</math> solution on a tile.</p>	Violet colouration	Presence of phenol
<p><b>b. Phthalein Reaction:</b> About 5 mg of the substance is heated with about 10 mg of phthalic anhydride and a drop of con. sulphuric acid in a semimicro tube. The solution is cooled and diluted with about 1 ml of water. A drop of the solution is mixed with a drop of 50%, sodium hydroxide solution on a tile.</p>	Red, blue or green color	Presence of phenol
<p><b>c. Liebermann's reaction :</b> About 5 mg of the substance is heated with about 5 mg of sodium nitrite and 2 drops of con. sulphuric acid in a semi micro tube. It is cooled and diluted with about 1 ml of water. A drop of it is placed on a tile and mixed with a drop of 10% sodium hydroxide solution.</p>	A bluish green color is produced	Presence of phenols
<p><b>4. Test for Carbohydrates:</b> (Molisch's test) A drop of an alcoholic solution of <math>\alpha</math>- naphthol is mixed with a drop of aqueous solution of the substance on a tile and was mixed with a drop of con. Sulphuric acid.</p>	A deep violet color	Presence of sugars
<p><b>5a. Test for aldehydes / ketones:</b> Borsche's reagent test:</p>	Red orange precipitate	

A drop of the substance ( if it is liquid) or a drop of an alcoholic solution is placed on a tile. A drop of the con. Solution of 2,4- DNP (Borsch reagent) is added to it.		Presence of aldehydes/ ketones
<b>5b. Schiff's reagent test:</b> A drop of the substance is mixed with a drop of Schiff's reagent	Pink colour	Presence of aldehyde
<b>5c.</b> 2 drops or about 5 mg of the substance is mixed with 2 drops of Fehling A and 2 drops of Fehling B solutions in a semimicro test tube. It is heated in a water bath for about 5 minutes.	Red precipitate	Presence of aldehyde/ Reducing sugars
<b>6a. Test for ester:</b> About 2 drops of the substance is heated with 2 drops of methanolic solution of hydroxylamine hydrochloride and 2 drops of 50% NaOH and cooled. A drop of it is placed on a tile. It is mixed with dil. HCl and neutral FeCl <sub>3</sub> , a drop of it each.	Violet color	Presence of ester
6b. drops of the substance is heated with 2 drops of 50% NaOH solution, till it is dissolved. Cool it and then add 4 drops of 50% HCl.	White precipitate	Presence of ester
<b>7a. Test for alcohols:</b> Add a small piece of Na metal to 2 drops of the substance.	Effervescence	Alcohols present
<b>7b.</b> A drop of the substance is added to a drop of acetyl chloride in fume hood.	Violent reaction with evolution of fumes.	Alcohols present

<p><b>8. Test for Hydrocarbons:</b> With 5mg each of the substance and picric acid prepare a saturated solution in alcohol separately (add 3 or 4 drops of alcohol). Mix The solutions, heat and Cool.</p>	Yellow precipitate	Presence of hydrocarbons

## II. Under C, H, (O), N group:

<p><b>1. Test for primary Amines :</b> <b>Dye test :</b> A drop of the substance is placed on a tile. A drop of dil. hydrochloric acid is added to it. A drop of saturated sodium nitrite solution followed by a drop of <math>\beta</math> - naphthol dissolved in 25% NaOH is added to it.</p>	Red azo dye	Presence of aromatic primary amine
<p><b>2. Test for Secondary Amines:</b> 2 drops of substance is mixed with 2 drops of dil. Hydrochloric acid and 2 drops of saturated solution of sodium nitrite in a semimicro tube. A drop of con. sulphuric acid and a drop of phenol are added. Heated for a minute. A drop of this solution is mixed with a drop of dil. NaOH on a tile.</p>	A bluish green color is produced	Presence of secondary amine
<p><b>3. Test for tertiary amine</b> <b>Dye test :</b> 2 drops of benzaldehyde and 4 drops of the substance are heated with 1 drop of con. sulphuric acid and 50 mg of lead dioxide. A drop of this solution is mixed with a drop of dil. HCl on a tile.</p>	Malachite green dye is formed	Presence of tertiary amine.
<p><b>4a. Test for nitro group:</b> <b>Reduction to amines :</b></p>		

<p>About 3 drops of the substance is reduced with 3 drops of con. hydrochloric acid and metallic tin or zinc in a semimicro tube, by heating for about 5 minutes. with a drop of the supernatant liquid dye test is performed on a tile.</p> <p>(as under test for amines)</p>	Red azo dye	Presence of nitro group
<p><b>4b. Mulliken - Barker's test:</b> About 3 drops of the substance and 3 drops of alcohol are boiled with 3 drops of calcium chloride solution and a pinch of zinc dust. Heated to boiling and cooled. A drop of it is mixed with a drop of Tollen's reagent on a tile.</p>	A black precipitate	Presence of nitro group
<p><b>5. Test for Anilide:</b> About 10 mg of the substance is heated strongly in a dry semi micro tube with soda lime. The resulting vapour is collected in another semi micro tube containing 5 drops of dil. hydrochloric acid. With a drop of the acid solution dye test is performed</p>	Scarlet Red dye	Presence of anilide
<p><b>6. Biuret test:</b> About 5 mg of the substance is heated strongly in a dry semi micro tube to its melting point. Cooled and the residue is dissolved in 3 drops of water. A drop of it is mixed with a drop of dil. CuSO<sub>4</sub> and with a drop of dil. NaOH solution on a tile.</p>	A violet colour	Presence of diamide
<p><b>7. Test for Aromatic monoamide:</b> About 5 mg of the substance is heated with 5 drops of 25% NaOH solution and cooled. A</p>	White precipitate	Presence of monoamide

drop of it is mixed with a drop of con. HCl		
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### III. Under C, H, (O), N, S group:

<p><b>1a. Test for thiourea:</b> About 5 mg of the substance is heated with 5 drops of dil. NaOH solution and cooled. A drop of it is mixed with a drop of lead acetate solution on a tile.</p>	A black / brown precipitate	Presence of thiourea
<p><b>1b.</b> 5 mg of the substance is heated in a dry semi micro tube until it melts. It is cooled and the residue is dissolved in 4 drops of water. A drop of it is mixed with a drop of neutral <math>\text{FeCl}_3</math> solution on a tile.</p>	Blood red coloration	Presence of thiourea